Materials

Ion-Exclusion Chromatography for Analyzing Organics in Water

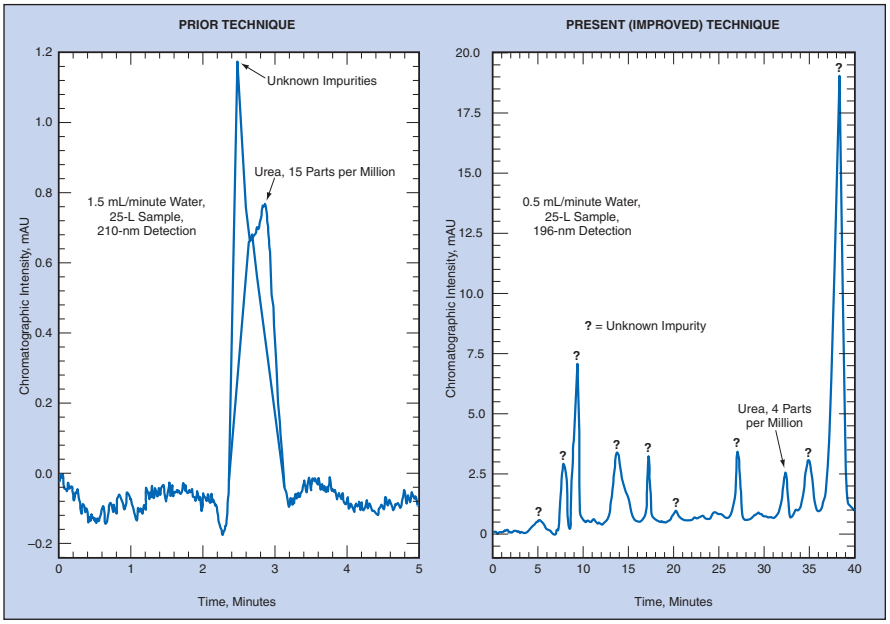
Resolution of nonvolatile organic compounds is increased significantly.

Lyndon B. Johnson Space Center, Houston, Texas

A liquid-chromatography technique has been developed for use in the quantitative analysis of urea (and of other nonvolatile organic compounds typically found with urea) dissolved in water. The technique involves the use of a column that contains an ion-exclusion resin; heretofore, this column has been sold for use in analyzing monosaccharides and food softeners, but not for analyzing water supplies.

The prior technique commonly used to analyze water for urea content has been one of high-performance liquid chromatography (HPLC), with reliance on hydrophobic interactions between analytes in a water sample and long-chain alkyl groups bonded to an HPLC column. The prior technique has proven inadequate because of a strong tendency toward co-elution of urea with other compounds. Co-elution often causes the urea and other compounds to be crowded into a narrow region of the chromatogram (see left part of figure), thereby giving rise to low chromatographic resolution and misidentification of compounds. It is possible to quantitate urea or another analyte via ultraviolet- and visible-light absorbance measurements, but in order to perform such measurements, it is necessary to dilute the sample, causing a significant loss of sensitivity.

The ion-exclusion resin used in the improved technique is sulfonated polystyrene in the calcium form. Whereas the alkyl-chain column used in the prior technique separates compounds on the basis of polarity only, the ion-exclusion-resin column used in the improved technique separates compounds on the basis of both molecular size and electric charge. As a result, the degree of separation is increased: instead of being crowded together into a single chromatographic peak only about 1 to 2 minutes wide as in the prior technique, the chromatographic peaks of different compounds are now separated from each other and spread out over a range about 33 minutes wide (see right part of figure), and the urea peak can readily be distinguished from the other peaks.



These **Chromatograms** were made from samples of space-shuttle humidity condensate, which contained dissolved urea and other impurities.

Although the analysis takes more time in the improved technique, this disadvantage is offset by two important advantages:

- Sensitivity is increased. The minimum concentration of urea that can be measured is reduced (to between 1/5 and 1/3 of that of the prior technique) because it is not necessary to dilute the sample.
- The separation of peaks facilitates the identification and quantitation of the various compounds. The resolution of the compounds other than urea makes it possible to identify those compounds by use of mass spectrometry.

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Selective Plasma Deposition of Fluorocarbon Films on SAMs

The fluorocarbon films are useful as etch masks and perhaps as dielectric layers.

Goddard Space Flight Center, Greenbelt, Maryland

A dry plasma process has been demonstrated to be useful for the selective modification of self-assembled monolayers (SAMs) of alkanethiolates. These SAMs are used, during the fabrication of semiconductor electronic devices, as etch masks on gold layers that are destined to be patterned and incorporated into the devices. The selective modification involves the formation of fluorocarbon films that render the SAMs more effective in protecting the masked